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Kevin D. McCarthy

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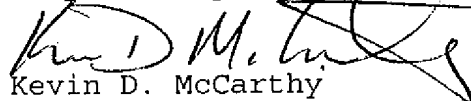
Dear Sir/Madam:

**Preliminary Amendment**

Applicant acknowledges on the transmittal sheet that this application is a continuation-in-part of PCT application number PCT/IL2005/000554, filed on May 30, 2005. Applicant has prepared a preliminary amendment to highlight those portions that have been (a) deleted from the original publication of the PCT application – bracketed material with a strikethrough – and (b) added to the original publication of the PCT application – bolded, italicized and underlined (to differentiate those sections that were underlined and bolded in the original PCT application) and tables IV and V. We enclosed an entire copy of the application in red-lined format since there are numerous changes and identified the paper as the red-lined version.

To assist the examiner, we have also enclosed a clean copy of the pending CIP application.

Respectfully submitted

  
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## **SCORCH PREVENTION IN FLEXIBLE** **POLYURETHANE FOAMS**

### **Field of the Invention**

5 The present invention relates to the prevention of discoloration in flexible polyurethane foams, a phenomenon commonly referred to as "scorching". More particularly, the invention relates to novel compositions useful to alleviate or prevent the aforementioned undesirable effect.

### 10 **Background of the Invention**

Scorching is an undesirable discoloration phenomenon which occurs [the] **within polyurethane (PU)** foam blocks, causing them to assume a yellow to brown color. This discoloration is especially apparent in the center of the blocks where the internal temperatures remain high for a relatively long period of time. The exposure of the interior of the foams to high temperatures leads to embrittlement and the core discoloration commonly known as scorching. In extreme cases this can result in ignition of the foam buns, sometimes with disastrous consequences. In some severe cases, it can cause a degradation of physical properties or, where scorching is particularly intense, can result in spontaneous combustion of freshly made foam blocks.

Flame retardants (**FR**), with few exceptions, [e.g., pentabromodiphenyl oxide,] exacerbate the "scorch" problems that arise during processing of **PU** [low density] foams. [It is known that the use of flame retardants (FR) in polyols can lead to even higher discoloration in polyurethane (PU) foams than the non FR grades.] Flame retardants[, with few exceptions,] create **several** [two major] problems for the manufacturers of water-blown, flexible, slab stock [foam. They increase the] **foams, including** **increased** "scorch" [problems that arise] during processing [of low

density foams] and [they increase the] **increased** smoldering tendency of foams subjected the California TB 117 standard.

[The flame retardant tribromoneopentyl alcohol (known as FR-513 ex  
5 Dead Sea Bromine Group (DSBG)) was investigated as flame retardant in  
PU foams already in 1975 [J.H. Botkin, Scorch Inhibitors for  
Polyurethane Slabstock Foams, Adv. Urethane Sci. Technol., vol. 14, pp.  
57-80, 1998].]

10 [The use of antioxidants has been proposed in the art and it can mitigate to some  
extent the increased scorch and discoloration in flame retarded polyols and  
foams. However, the literature consulted, and experiments carried out, by the  
inventors indicate that antioxidants by themselves are not very efficient in  
preventing scorch when halogen-containing flame retardants, are present in the  
15 formulation. Thus, the art has so far failed to provide an efficient solution to the  
problem of scorch taking place during the manufacturing of flexible polyurethane  
foams.]

**Antioxidants have been proposed for use in flame retarded foams,**  
20 **but by themselves they are not very efficient. For example when**  
**halogen containing FR are present in the formulation. The art has**  
**so far failed to provide a generally efficient solution to the problem**  
**of scorch taking place during the manufacturing of flexible**  
**polyurethane foams comprising any FR selected from those used in**  
25 **the art.**

**It is therefore an object of the present invention to provide an anti-**  
**scorch composition addressing the problem of scorch for flame-**  
**retarded flexible polyurethane formulations.**

30

It is **another** object of the present invention to provide an anti-scorch  
composition that efficiently addresses the problem of scorch for flexible

polyurethane formulations flame retarded with scorch-inducing FR such as phosphorus-based FR or halogen-containing FR, whether aliphatic or aromatic halogenated FR, or their combination.

- 5 It is still another object of the invention to provide an anti-scorch combination that overcomes the drawbacks of the prior art.

It is yet another object of the invention to provide a method for preventing or diminishing the occurrence of scorch during the manufacturing of  
10 flexible polyurethane foams.

Other purposes and advantages of the invention will become apparent as the description proceeds.

15 **Summary of the Invention**

The invention provides ~~[is directed to]~~ an anti-scorch composition for flame-retarded flexible polyurethane foams, comprising ~~[, in combination, one or more]~~ at least one antioxidant agent, ~~[together with one or more salt(s) of an organic acid]~~ an epoxy compound, and at least one  
20 organic phosphite alone or in combination with at least one metal salt of a monocarboxylic or dicarboxylic acid. ~~[According to a preferred embodiment of the invention the]~~ Said organic acid is selected from among saturated or unsaturated, aliphatic or aromatic, and monocarboxylic or dicarboxylic organic acids. ~~[According to another preferred embodiment of the invention the salt of the organic acid is a salt]~~ Said metal salt is selected from the group consisting of salts of Ca, Zn, Ba, and Sn. An illustrative example of such salts may include barium oleate, barium t-butylbenzoate, barium ethylhexanoate, zinc t-butylbenzoate, calcium dimethyl  
25 ethylbenzoate, etc. Said at least one antioxidant agent, i.e. one or more compounds, may be selected from among phenols and amino  
30

oxygen scavengers, and their mixtures. Said phenol is preferably a hindered phenol, and said amino oxygen scavenger is preferably an alkylated diphenylamine. In a preferred embodiment of the invention, said at least one antioxidant agent comprises a mixture of hindered phenol and an alkylated diphenylamine. The flame-retarded foams of the invention may be retarded by any FR known in the art. In a preferred embodiment of the invention, the PU foams are retarded with phosphorus-based flame retardant (FR). In another embodiment, said flame-retarded foams are retarded with a halogen-containing flame retardant, e.g. with an aliphatic or aromatic brominated or chlorinated FR. Non-limiting examples of FR possibly used in the composition of the invention include brominated epoxy, tribromoneopentyl alcohol, tris(2-chloroisopropyl) phosphate, chlorinated alkylphosphate ester, halogenated aryl esters/aromatic phosphate blend, and pentabromobenzyl alkyl ethers. The composition with reduced scorch phenomenon according to this invention comprises an epoxy compound, which may be, for example, selected from among diglycidyl ether of bisphenol A and its derivatives. Said epoxy compound, added to the mixture of the invention, has available epoxy groups, i.e., epoxy groups essentially not neutralized by previous curing. Said organic phosphite may be, for example, selected from among tris(alkylphenyl) phosphites, trialkyl phosphites, dialkyl phenyl phosphites, alkyl diphenyl phosphites, and triphenyl phosphites.

The invention further provides a method for preventing or diminishing scorch in a flame-retarded flexible polyurethane foam, comprising adding to the polyurethane composition, prior to foaming, at least one antioxidant agent, an epoxy compound, and an organic phosphite alone or with at least one metal salt of a monocarboxylic or dicarboxylic acid. Illustrative and non-limitative examples of antioxidant agents are phenols and amino oxygen

scavengers, such as hindered phenols. Illustrative and non-limitative examples of amino oxygen scavengers include alkylated diphenylamines. The flame-retardant may be a phosphorus-based flame retardant, and/or halogen-containing flame retardant, e.g. an aliphatic or aromatic brominated or chlorinated FR. According to a further preferred embodiment of the invention the composition further comprises an epoxy compound, such as - but not limited to - diglycidyl ether of bisphenol A and its derivatives.

10 ~~[Illustrative and non-limitative examples of antioxidant agents are phenols and amino oxygen scavengers, such as hindered phenols.]~~

~~[Illustrative and non-limitative examples of amino oxygen scavengers include alkylated diphenylamines.]~~

15

~~[According to a preferred embodiment of the invention the flame-retardant is a halogen-containing flame-retardant, such as - but not limited to - tribromopentyl alcohol.]~~

20 ~~[In another aspect the invention is directed to a method for preventing or diminishing scorch in flame-retarded flexible polyurethane foams, comprising adding to the polyurethane composition, prior to foaming, one or more antioxidant agents, together with one or more salt(s) of an organic acid, as hereinbefore defined.]~~

25

The above and other objects and advantages of the invention will be better understood from the following illustrative and non-limitative examples.

#### **[Test Procedure:]**

30

#### **Examples 1-15**

Microwave (MW) test protocol for scorch evaluation

The test method consists of the following steps:

1. Foam production in a small shoe box with a square cross-section.
2. Immediately after the foam rise is complete (usually less than 2 minutes), the foam is heated using a microwave (MW) oven using a predetermined time and power level.
- 5 3. The foam is then heated in an oven at  $120 \pm 2^\circ\text{C}$  for 2 min.
4. The foam is allowed to cure at RT (room temperature) for an additional 15 min.
5. By the end of the RT curing time the foam is cut (usually vertically) and the scorch is observed.

10

#### Scorch evaluation

The scorch is evaluated using two methods:

1. The foam is photographed using a digital camera. This allows for a visual comparison between the scorch of a reference formulation and the  
15 scorch of the formulation under investigation.
2. The foam color is analyzed using a spectrophotometer. The results are expressed in the usual color space:  $L^*a^*b$  and performed accordingly to ASTM D-2244.

Note: The scorch intensity may change from day to day according to the  
20 physical conditions of the surroundings in which the foam has been prepared (usually the temperature and relative humidity). This is why it is customary to prepare each day a reference sample.

#### [Example]

#### Foam preparation

- 25 The mixture was prepared in a 0.65 l disposable cup. The components were added one at a time starting with the polyol. The mixture was vigorously mixed at 3500 rpm for 10 seconds following the addition of each component, not including the Toluene diisocyanate (TDI). After the addition of TDI, the mixture was mixed for an additional 10 sec and then  
30 poured into a 25X25X17 cm cardboard box. The times between the TDI



addition and the pouring into the cardboard box and the end of the foam blowing (rise time) were monitored.

#### Comparative samples and results

5 The effect of the antiscorching ingredients and their combinations was measured on the darkest areas on the foam after microwave oven treatment, using a spectrophotometer which provides color measurements expressed in the L\*a\*b color space. The most relevant color parameters for scorch assessment are  $\Delta b$  and  $\Delta E$ .

10

The color parameters are given as normalized values relative to the reference specimens. As explained in the MW oven procedure, this particular method for scorch propensity assessment requires that a new reference foam be prepared, subjected to MW oven procedure and  
15 measured in each and every day of measurements. This requirement is related to the effect various experimental conditions, such as the temperature and the relative humidity of the air in the lab may have on the level of scorch. The normalized  $\Delta b$  and  $\Delta E$  differences between a reference foam (containing no antiscorching ingredients) and foams  
20 containing various ingredients with antiscorch effects, are calculated as follows:

$$\Delta\Delta b(\text{normalized}) = \frac{\Delta b(\text{reference}) - \Delta b(\text{sample})}{\Delta b(\text{reference})} \cdot 100$$

$$\Delta\Delta E(\text{normalized}) = \frac{\Delta E(\text{reference}) - \Delta E(\text{sample})}{\Delta E(\text{reference})} \cdot 100$$

25 Note: A value greater than 100 can sometimes emerge from these calculations since both  $\Delta b$  and  $\Delta E$  for each specimen are compared to a factory white standard. The higher the  $\Delta\Delta b$  and  $\Delta\Delta E$  values, the lower the scorch.

Formulations for two grades of foams are shown in Tables I and II: Medium density foams (Table I) have a density of approximately 25 Kg/m<sup>3</sup>; Low density foams (Table II) have a density of approximately 15 Kg/m<sup>3</sup>. The component amounts in tables I, II, IV, and V are expressed as weight parts relative to 100 parts of polyol (phr).

Ingredients: AO1 and AO5 are antioxidants produced by Goldschmidt (Degussa) and contain combinations of hindered phenols and aromatic diamines.

Epoxy 828 is Diglycidyl ether of bisphenol A (DGEBA).

ESBO = Epoxidized Soya Bean oil.

All other ingredients below the FR-513 line in the table are metallic salts (Ca, Zn, Ba, Sn) of organic acids, and organic phosphites.

Table I Anti-scorch performance of medium [Normal] density foams

	1	2	3	4	5	6	7	8	9
Polyol	100	100	100	100	100	100	100	100	100
Water	4.2	4.2	4.2	4.2	4.2	4.2	4.2	4.2	4.2
Silicon 8228	1.1	1.1	1.1	1.1	1.1	1.1	1.1	1.1	1.1
Amine Dabco 33LV	0.09	0.09	0.09	0.09	0.09	0.09	0.09	0.09	0.09
Amine BDE	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03
Tin T-9	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25
Toluene di-isocyanate (TDI)	56.31	56.31	56.31	56.31	56.31	56.31	56.31	56.31	56.31
FR-513	5	5	5	5	5	5	5	5	5
AO5	0.42	0.21	0.85						
Calcium stearate (CaSt)	0.42	0.21							
PK4610	0.42	0.21							
Epoxy 828			0.85		0.1	0.1	0.21		
ESBO				1.7					
LZB 138					0.21			0.21	0.21
AO 1					0.21	0.21	0.21	0.21	0.21
CZ400						0.21		0.21	
CZ 118 S							0.21		0.21
ΔΔb	90.13	106.1	49.17	-	82.32	77.06	93.93	84.58	102.9

				132.6					
$\Delta\Delta E$	17.04	10.84	18.34	- 99.43	27.61	32.67	30.14	37.42	33.98

Table II *Anti-scorch performance of* low density foams

	<u>10</u> [1]	<u>12</u> [2]	<u>12</u> [3]	<u>13</u> [4]	<u>14</u> [5]	<u>15</u> [6]
Polyol	100	100	100	100	100	100
Water	5.25	5.25	5.25	5.25	5.25	5.25
Silicon 8228	1.0	1.0	1.0	1.0	1.0	1.0
Amine Dabco 33LV	0.06	0.06	0.06	0.06	0.06	0.06
Amine BDE	0.046	0.046	0.046	0.046	0.046	0.046
Tin T-9	0.37	0.37	0.37	0.37	0.37	0.37
Methylene Chloride (MeCl)	11	11	11	11	11	11
TDI	71.1	71.1	71.1	71.1	71.1	71.1
FR-513	15	15	15	15	15	15
AO5			0.1	0.3		0.2
Epoxy 828	0.5	1.0				
BM270					1.0	0.2
LZB287						0.6
$\Delta\Delta b$	-14.11	24.37	-15.10	-77.1	95.29	50.90
$\Delta\Delta E$	0	13.73	-10.13	-41.03	27.80	11.70

Table III Composition of ingredients used as antiscorch materials.

Producer	Name	Ingredients		State
Akros	Lankromark LZB287	1) Barium oleate Barium t-butylbenzoate	40%	Liquid
		2) Zinc 2-ethylhexanoate	5-10%	
		3) Phosphite esters	20-40%	
		4) 2-(2-butoxyethoxy)ethanol	5-10%	
		5) Phenol	5-10%	
	Lankromark LZB413	1) Barium 2-ethylhexanoate Barium oleate Barium t-butylbenzoate	20%	Liquid
		2) Zinc 2-ethylhexanoate	1-5%	
		3) Zinc t-butylbenzoate	1-5%	
		4) Phosphite esters	20-40%	
		5) Trisnonylphenyl phosphate	1-5%	

		6) 2-(2-butoxyethoxy ethanol)		
	Lankromark LZB138	1) Barium oleate Barium t-butylbenzoate	~30%	Liquid
		2) Zinc 2-ethylhexanoate	10-20%	
		3) Phosphite esters	20-40%	
		4) 2-(2-butoxyethoxy) ethanol	5-10%	
		5) Phenol	1-5%	
	Lankroflex E2307	1) Epoxidized soya bean oil	> 99%	Liquid
	Tinstab BTS71S	1) n-Butyltin tris (2-ethylhexylthio- glycolate)	~20 %	Liquid
		2) Di-n-butyltin bis (2-ethylhexylthio- glycolate)	~70%	
	Tinstab BM270	1) Di-n-butyltin bis (methylmaleate)	~95%	Liquid
Shell	Epoxy 828		~100%	Liquid
Chemson Group	Naftosafe PK4610	Ca/Zn organic stabilizer	-	Powder
Gold- schmidt	Ortegol AO1	Steric hindered phenol derivate	66.7%	
		Alkylated diphenylamine	33.3%	
	Ortegol AO5	Steric hindered phenol derivate	70-72%	
		Alkylated diphenylamine	20-22%	
Crompto n	Mark CZ 400 (Ca/Zn stabilizer)	2-(2-Butoxyethoxy)ethanol	<10%	Liquid
		Alkylarylphosphites	55-65%	
		Calcium 4-(1,1-dimethylethyl)benzoate	<10%	
	Mark CZ 118S (Ca/Zn stabilizer)	Tris(nonylphenyl)phosphate	30-40%	Liquid
		Other components - not listed		
	Mark BZ 592 (Ba/Zn stabilizer)	Solvent naphta, light aromatic	<10%	Liquid
		Barium compounds	35-45%	
		Triisodecyl phosphate	<25%	

Producer	Name	Ingredients		State
		Solvent naphta, light aromatic	<10%	
		Alkylarylphosphites	20-30%	
		Zinc bis(p-nonylphenolate)	<3%	

Crompton	Mark BZ 562 (Ba/Zn stabilizer)	Barium compounds	35-45%	Liquid
	Mark BZ 555 (Ba/Zn stabilizer)	Tris(2-ethylhexylmercaptoacetate) phosphate	5-15%	Liquid
		Diisodecyl phenyl phosphate	25-35%	
		Solvent naphta, light aromatic	5-15%	
		Barium compounds	20-30%	
	Mark BZ 563 (Ba/Zn stabilizer)	Diisodecyl phenyl phosphate	30-50%	Liquid
		2-(2-Butoxyethoxy)ethanol	<5%	
		Zinc bis(p-nonylphenolate)	<5%	
		Barium compounds	20-30%	

From the results in Tables I and II it is clearly seen that the compositions of the invention attain a substantial improvement.

- 5 The notations and composition of materials used as antiscorch ingredients in the examples in Tables I and II are detailed in Table III.

Example 3 in Table I and Examples 12 and 13 in Table II, respectively, demonstrate that the use of a conventional antioxidant, i.e. a mixture of hindered phenols and alkylated diphenyldiamines, alone or together with an epoxy moiety, does not prevent scorch in a bromine-containing FR formulation. Epoxy alone, either as DGEBA or ESBO, are not effective either (Examples 4 in Table 1, and Examples [1 and 2] **10 and 11** in Table II). However, addition of organic phosphites alone or in  
15 combination with metallic salts of various organic acids, significantly improves the resistance to scorch of a polyurethane flexible foam formulation including a bromine-containing flame retardant (Examples 8, 9 - Table I, and Examples [5,6] **14, 15** - Table II).

Examples 16-25

MW test protocol for scorch evaluation

1. Foam production in a small shoe box with a square cross-section.

5 2. Immediately after the foam rise is complete (usually less than 2 minutes), the foam is heated in a microwave (MW) oven that is equipped with an electronic controlling circuit that controls the overall heating energy emitted by the MW oven. Controlling feedback is accomplished via measuring the temperature of a given  
10 constant mass of water co-heated alongside the foam. The water temperature closely follows a pre-set rate of temperature rise (ramp) over a predetermined period of time so that a constant temperature difference is maintained in each heating cycle.

15 3. The foam is then placed in an oven at 110-120°C for 17 minutes not only to cure but also to slow down the foam's natural cooling and to isolate it from climate and surrounding changes as well. The foam's core temperature is being monitored throughout this stage with a k-type 1.6 mm diameter thermocouple plugged into a data-logger.

20 4. Following the 17 minutes curing the foam is sliced perpendicular to foam rise direction, obtaining square cross-section slices. The surface of the central slice is then photographed and its scorch level is evaluated.

25 Digital scorch evaluation

The effect of anti-scorching ingredients on various scorch-forming halogenated flame retardants in various common flexible polyurethane formulations was measured all over the surface of the central foam slice, where the foam core temperature was the  
30 highest along the disclosed procedure, rather than locally on selected darkest zones alone. Digital photographs taken under fully controlled parameters (illumination, filming angles) were processed by graphic software that scans the photographs having

the same resolution and sums up the number of pixels having different colors over a selected slice area. The resulting value, Unique Colors (UC), highly correlates with the visual comparative inspection of scorch intensity. The UC values are straightforward, the lower they are the less scorched is the foam. A reduced UC value indicates a better anti-scorch effect.

Note: the controlled emission of the MW energy during the foam heating stage of the test procedure, together with the controlled and slowed down cooling of the foam that is isolated from the surrounding climate changes, were statistically proved to accomplish highly reproducible day-to-day results. Scanning to measure the entire scorched area also was well contributing to the accuracy and reproducibility of the test procedure. Nevertheless, often a reference formulation was carried as an internal test to assure that test parameters are fully kept and the resulting scorch intensity falls within the narrow variability limits of the procedure.

#### Foam preparation

The foam mixture was prepared in a 0.65 l disposable cup. All components, but the water and the toluene di-isocyanate (TDI), were successively added starting with the polyol. The mixture was vigorously mixed at 4400 rpm for 15 seconds, followed by the injection of pre-weighed water under continuous mixing. Mixing was allowed for 20 seconds more before the pre-weighed TDI was introduced, followed by 10 seconds of further mixing and an immediate pouring of the mixture into a 25x25x17 cm<sup>3</sup> cardboard box. Foam rise time, from pouring to blow-off, was recorded.

Various halogen-containing common commercial flame retardants, among them some well known to be accompanied by an intense scorching process, were introduced into the foam formulations. The marked effect of the combination of the anti-scorching components

was demonstrated by comparing the measured scorch intensity (UC values) with and without the presence of each of these flame retardants in the formulation. These results and the normal density formulation (foam density ca. 25 Kg/m<sup>3</sup> in FR-513 containing foams) used with the various flame retardants - with the necessary changes of TDI - is presented in table IV. The efficacy of the same combination of anti-scorching components toward various halogen-containing flame retardants is shown to be equally high for different foam formulations or foam densities, as presented in table V.

A combination of anti-scorch ingredients according to the invention, comprising hindered phenol, alkylated diphenylamine, epoxy compound, and organic phosphites with or without metallic salts of organic acids, is demonstrated hereafter to effectively diminish or prevent the scorch occurrence within foams containing scorch-forming flame retardants, such as: halogenated - wherein the halogen is either aromatic or aliphatic - and/or halogenated phosphates - wherein the halogen is either aromatic or aliphatic - and/or phosphorus-based flame retardants. Illustrative and non-limitative examples of such flame retardants are: tribromoneopentyl alcohol (FR-513 ex Dead Sea Bromine Group, DSBG), tris(2-chloroisopropyl) phosphate (TCPP, Fyrol FR-2 ex Akzo Nobel), chlorinated alkylphosphate ester (Amgard V6 ex Albright and Wilson/ Antiblaze V6 ex Albemarle), Halogenated aryl esters/Aromatic phosphate Blend (FM-550 ex Great Lakes), pentabromobenzyl alkyl ethers (FR-1435X ex DSBG), brominated epoxy F-3014 (ex DSBG) dissolved in phosphates (Phosflex 31L ex Akzo Nobel) 1:1 w/w.

The selected combination of the anti-scorch ingredients comprises the materials:



**AO5: Antioxidant produced by Goldschmidt (Degussa), containing a combination of hindered phenols and aromatic diamines.**

**Epoxy 828: EPON™ Resin 828 produced by Resolution Performance Products is Diglycidyl ether of bisphenol A (DGEBA).**

5 **Metallic (Ba, Zn, Ca or Sn) salts of organic acids and organic phosphites are included in Baerlocher™ product.**

10 **Table IV Anti-scorch performance of medium density foams with halogenated or phospho-halogenated flame retardants**

Ingredient	16	16a	17	17a	18	18a	19	19a	19b	20	20a	21	21a
Flame retardant	Fyrol FR-2 (TCPP)		Amgard V6 (Antiblaze V6)		F-3014 & Phosphates		FR-513			FM 550		FR-1435X	
	15		10		13		8			14		14	
Polyol	100		100		100		100			100		100	
Silicon Nix L-650	0.95		0.95		0.95		0.95			0.95		0.95	
Amine Dabco 33LV	0.018		0.018		0.018		0.018			0.018		0.018	
Amine BDE	0.0624		0.0624		0.0624		0.0624			0.0624		0.0624	
Tin T-9	0.153		0.153		0.153		0.153			0.153		0.153	
Water	4.63		4.63		4.63		4.63			4.63		4.63	
Toluene di-isocyanate (Index 119)	62.03		62.03		62.53		64.59			62.03		62.03	
Anti-scorch Combinat. 1 <sup>a</sup>	0.0	1.0	0.0	1.0	0.0	1.0	0.0	1.0	-	0.0	1.0	0.0	1.0
Anti-scorch Combinat. 2 <sup>b</sup>									1.0				
Scorch level (UC)	2340 8	492 7	4318 8	777 7	2823 8	613 4	2741 4	5640 4	5238 4	711 1	595 0	780 6	595 6
Scorch reduction, %		-79		-82		-78		-79	-81		-16		-24

15 <sup>a</sup> A combination of anti-scorch components that contains an anti-oxidant, epoxy, organic phosphites and metal salts of organic acids.

<sup>b</sup> A combination of anti-scorch components that contains an anti-oxidant, epoxy and organic phosphites.

- 5 **The anti-scorch combination (AS package) demonstrated in Table IV is shown to maintain its high efficacy in diminishing or preventing scorching in foams containing scorch-forming flame retardants independently of the foam formulation, or the foam densities (e.g. normal – ca. 25 kg/m<sup>3</sup> or low – ca. 15 kg/m<sup>3</sup>, in FR-513 containing**  
 10 **foams). These results are presented in table V.**

- 15 **Table V Anti-scorch performance of medium and low density formulations with different FRs**

Ingredient	22	22a	23	23a	24	24a	25	25a
	Normal density foams - Second formulation				Low density foams			
Flame retardant	Fyrol FR-2 (TCPP)		FR-513		Fyrol FR-2 (TCPP)		FR-513	
	15		6.5		15		8	
Polyol	100		100		100		100	
Silicon Niox L-650	1.1		1.1		1.0		1.0	
Amine Dabco 33LV	0.09		0.09		0.06		0.06	
Amine BDE	0.03		0.03		0.047		0.047	
Tin T-9	0.25		0.25		0.37		0.37	
MeCl <sub>2</sub>	-		-		11.0		11.0	
Water	4.20		4.20		5.25		5.25	
Toluene di- isocyanate (Index 114)	54.68		56.67		66.27		68.72	
Anti-scorch Combinat. 1 <sup>a</sup>	0.0	1.0	0.0	0.81	0.0	1.0	0.0	1.0
Scorch level (UC)	2453 7	475 8	2366 7	570 6	815 9	542 1	791 0	470 0
Scorch reduction, %		-81		-76		-34		-41

<sup>a</sup> A combination of anti-scorch components that contains an anti-oxidant, epoxy, organic phosphites and metal salts of organic acids.

5 Considering the background color measured over non-scorched areas, the selected anti-scorch package has been proved to significantly reduce and nearly to diminish scorch occurrence in flexible polyurethane foams containing scorch-forming flame retardants, independently of their formulation or density.

10 A clear improvement was also achieved in foams containing flame retardant that so far were considered by foam producers to produce an acceptable scorch level, such as the case of FM 550.

15 All the above description has been provided for the purpose of illustration, and is not intended to limit the invention in any way. Various modifications can be carried out in the method and system according to the invention, without departing from its spirit.

CLAIMS

1. (Currently Amended) An anti-scorch composition for flame-retarded flexible polyurethane foams, comprising ~~in combination, one or more antioxidant agents together with one or more salt(s) of an organic acid]~~ **at least one antioxidant agent, an epoxy compound, and at least one organic phosphite alone or in combination with at least one metal salt of a monocarboxylic or dicarboxylic acid.**
2. (Currently Amended) A composition according to claim 1, wherein said organic acid is selected from among saturated or unsaturated, aliphatic or aromatic ~~[mono- or di-carboxylated acids],~~ **and monocarboxylic or dicarboxylic organic acids.**
3. (Currently Amended) A composition according to claim 1, wherein the **metal in said metal** salt ~~[of the organic acid]~~ is **selected from the group consisting** ~~[a salt]~~ of Ca, Zn, Ba, and Sn.
4. (Currently Amended) A composition according to ~~[any one of claims 1 to 3]~~ **claim 1**, wherein ~~[the]~~ **said at least one** antioxidant **agent** ~~[agent(s)]~~ is selected from among phenols and amino oxygen scavengers.
5. (Original) A composition according to claim 4, wherein the phenol is a hindered phenol.
6. (Original) A composition according to claim 4, wherein the amino oxygen scavenger is an alkylated diphenylamine.

7. (Currently Amended) A composition according to claim 1, wherein ~~[the flame retardant is a halogen-containing flame retardant]~~ said at least one antioxidant agent comprises a mixture of hindered phenol and an alkylated diphenylamine.
  
8. (Currently Amended) A composition according to claim 1, ~~[7,~~ wherein the flame retardant is tribromoneopentyl alcohol] wherein said flame-retarded foams are retarded with an aliphatic or aromatic, phosphorus-based, flame retardant (FR).
  
9. (Currently Amended) A composition according to ~~[any one of~~ claims ~~1-8]~~ claim 1, wherein said flame-retarded foams are retarded with a halogen-containing flame retardant. ~~[further comprising an epoxy compound]~~
  
10. (Currently Amended) A composition according to claim 1 ~~[9,~~ wherein the epoxy compound is selected from among diglycidyl ether of bisphenol A and its derivatives] wherein said flame-retarded foams are retarded with an aliphatic or aromatic, brominated or chlorinated, FR.
  
11. (Currently Amended) A ~~[method for preventing or diminishing~~ scorch in a flame-retarded flexible polyurethane foam, comprising adding to the polyurethane composition, prior to foaming, one or more antioxidant agents, together with one or more salt(s) of an organic acid] composition according to claim 1, wherein said flame-retarded foams are retarded with a FR selected from the group consisting of tribromoneopentyl alcohol, tris(2-chloroisopropyl) phosphate, tris(dichloropropyl) phosphate, chlorinated alkyl phosphate ester, halogenated aryl

esters/aromatic phosphate blend, pentabromobenzyl alkyl ethers, and brominated epoxy.

12. (Currently Amended) A ~~[method according to claim 11, wherein the organic acid is selected from among saturated, aliphatic or aromatic mono or di-carboxylated acids]~~ composition according to claim 1, wherein said epoxy compound is selected from among diglycidyl ether of bisphenol A and its derivatives.

13. (Currently Amended) A ~~[method according to claim 12, wherein the salt of the organic acid is a salt of Ca, Zn, Ba or Sn]~~ composition according to claim 1, wherein said organic phosphite is selected from among tris(alkylphenyl) phosphites, trialkyl phosphites, dialkyl phenyl phosphites, triphenyl phosphites, and alkyl diphenyl phosphites.

14. (Currently Amended) A method ~~[according to claims 11 to 13, wherein the antioxidant agent(s) is selected from among phenols and amino-oxygen-seavengers]~~ for preventing or diminishing scorch in a flame-retarded flexible polyurethane foam, comprising adding to the polyurethane composition, prior to foaming, at least one antioxidant agent, an epoxy compound, and organic phosphites alone or in combination with at least one metal salt of a monocarboxylic or dicarboxylic acid.

15. (Currently Amended) A method according to claim 14, wherein the ~~[phenol is a hindered phenol]~~ organic acid is selected from among saturated or unsaturated, aliphatic or aromatic, monocarboxylic or dicarboxylic organic acids.

16. (Currently Amended) A method according to claim 14, wherein the ~~[amino-oxygen-seavenger is an alkylated diphenylamine]~~ metal

in said metal salt is selected from the group consisting of Ca, Zn, Ba or Sn.

17. (Currently Amended) A method according to claim [11, wherein the flame-retardant is a halogen-containing flame-retardant] 14, wherein said at least one antioxidant agent is selected from among phenols and amino oxygen scavengers.
18. (Currently Amended) A method according to claim [17, wherein flame-retardant is tribromoneopentyl-alcohol] 14, wherein said phenol is a hindered phenol.
19. (Currently Amended) A method according to [any one of claims 11 to 18, further comprising adding an epoxy compound] claim 14, wherein said amino oxygen scavenger is an alkylated diphenylamine.
20. (Currently Amended) A method according to claim [19, wherein the epoxy compound is selected from among diglycidyl ether of bisphenol A and its derivatives] 14, wherein said at least one antioxidant agent comprises a mixture of hindered phenol and an alkylated diphenylamine.
21. (Currently Amended) A method [for preventing or diminishing seorch in a flame-retarded flexible polyurethane foam, substantially as described and illustrated] according to claim 14, wherein said flame-retarded foam is retarded with an aliphatic or aromatic, phosphorus-based, flame retardant FR).
22. (Currently Amended) A method according to claim 14, wherein said flame-retarded foam is retarded with a halogen-

containing flame retardant. [~~An anti-scorch composition for flame-retarded flexible polyurethane foams, essentially as described and illustrated and with particular reference to the examples]~~

23. (New) A method according to claim 14, wherein said flame-retarded foam is retarded with a brominated or chlorinated, aliphatic or aromatic, FR.
24. (New) A method according to claim 14, wherein said flame-retarded foam is retarded with a FR selected from the group consisting of tribromoneopentyl alcohol, tris(2-chloroisopropyl) phosphate, tris(dichloropropyl) phosphate, chlorinated alkylphosphate ester, halogenated aryl esters/aromatic phosphate blend, pentabromobenzyl alkyl ethers, and brominated epoxy.
25. (New) A method according to claim 14, wherein said epoxy compound is selected from among diglycidyl ether of bisphenol A and its derivatives.
26. (New) A method according to claim 14, wherein said organic phosphite is selected from among tris(alkylphenyl) phosphites, trialkyl phosphites, dialkyl phenyl phosphates, triphenyl phosphites, and alkyl diphenyl phosphites.



Abstract

Compositions are provided for alleviating or preventing discoloration, known as "scorching", in flame-retarded flexible polyurethane foams. The anti-scorch compositions contain combinations of antioxidant agents, epoxy compounds, organic phosphites - alone or in combination with metal salts of carboxylic acids. The compositions are useful, for example, for polyurethane foams retarded with aliphatic or aromatic phosphorus-based flame retardants, or with halogen-containing flame retardants. [An Anti-

~~scorch composition for flame retarded flexible polyurethane foams, comprising, in combination, one or more antioxidant agents, together with one or more salt(s) of an organic acid.]~~

# Exhibit B

**Kevin McCarthy - Your ref: 17660/US/04 CIP; Our ref: 0-06-172**

**From:** Kevin McCarthy  
**To:** Cynthia Field (L&L); Gilad Hadari (L&L); michal Perlmutter (L&L)  
**Date:** 8/25/2006 4:20 PM  
**Subject:** Your ref: 17660/US/04 CIP; Our ref: 0-06-172  
**Attachments:**

Dear Colleagues:

The USPTO returned our postcard that confirms the application was submitted to the USPTO on August 3, 2006 and accorded U.S. patent application serial number 10/588,398.

Please have the inventors execute the attached declaration and return it to us. If the executed declaration is returned to us prior to our receipt of the Notice of Missing Parts, we will file the executed declaration upon receipt of that document.

Kevin D. McCarthy  
 Roach Brown McCarthy & Gruber, P.C.  
 420 Main Street - 1620 Liberty Building  
 Buffalo, New York 14202  
 Phone 716-852-0400  
 Facsimile 716-852-2535  
 E-Mail [kdmccarthy@roachbrown.com](mailto:kdmccarthy@roachbrown.com)

U. S. Patent and Trademark Office

**IP15 Rec'd PCT/PTO 03 AUG 2006**

Inventor:	Bron, et al
Title:	Scorch Prevention in Flexible Polyurethane
Transmittal letter:	(3 pages)
Clean Copy to Amend:	(20 pages)
A2:	(18 pages)
A3:	(2 pages)
Declaration:	(Unsigned 3 pages)
IDS:	(5 pages)
Prelim. Amendment:	(24 pages)
Form 304:	(1 page)
Request:	(4 pages)
Form 2038:	\$900 (1 page)
Our Ref. No.:	<b>0-06-172</b>
Postcard:	Stamped, self-addressed

**10/588398**

# Exhibit C

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

**TRANSMITTAL LETTER TO THE UNITED STATES  
DESIGNATED/ELECTED OFFICE (DO/EO/US)  
CONCERNING A SUBMISSION UNDER 35 U.S.C. 371**

ATTORNEY'S DOCKET NUMBER

0-06-172

U.S. APPLICATION NO. (If known, see 37 CFR 1.5)

N/A

INTERNATIONAL APPLICATION NO.

PCT/IL2005/000554

INTERNATIONAL FILING DATE

May 30, 2005

PRIORITY DATE CLAIMED

June 10, 2004

TITLE OF INVENTION

Scorch Prevention in Flexible Polyurethane Foams

APPLICANT(S) FOR DO/EO/US

Bron et al.

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a submission under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a submission under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below.
4. ☒ The US has been elected (Article 31).
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
  - a. ☒ is attached hereto (required only if not communicated by the International Bureau).
  - b. ☐ has been communicated by the International Bureau.
  - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).
  - a. ☒ is attached hereto.
  - b. ☐ has been previously submitted under 35 U.S.C. 154(d)(4).
7. ☐ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
  - a. ☐ are attached hereto (required only if not communicated by the International Bureau).
  - b. ☐ have been communicated by the International Bureau.
  - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
  - d. ☐ have not been made and will not be made.
8. ☐ An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)). (unsigned - 3 pages)
10. ☐ An English language translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Clean Copy w/Amend - 20p.

A 2 - 18 pages

A 3 - 2 pages

Items 11 to 20 below concern document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98. (5 pages)
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A preliminary amendment. (24 pages)
14. ☐ An Application Data Sheet under 37 CFR 1.76.
15. ☐ A substitute specification.
16. ☐ A power of attorney and/or change of address letter.
17. ☐ A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 37 CFR 1.821-1.825.
18. ☐ A second copy of the published International Application under 35 U.S.C. 154(d)(4).
19. ☐ A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).

This application is a continuation-in-part of PCT application no. PCT/IL2005/000554, filed on May 30, 2005.

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

U.S. APPLICATION NO. (if known, see 37 CFR 1.5) <b>N/A</b>		INTERNATIONAL APPLICATION NO. <b>PCT/IL2005/000 554</b>		ATTORNEY'S DOCKET NUMBER <b>O-06-172</b>	
20. Other items or information: <b>Form 304 (1 page), Request (4 pages); postcard, Form 2038 for \$900 (1 page)</b>					
The following fees have been submitted				CALCULATIONS	PTO USE ONLY
21. <input checked="" type="checkbox"/> Basic national fee (37 CFR 1.492(a))..... \$300				\$ <b>300</b>	
22. <input checked="" type="checkbox"/> Examination fee (37 CFR 1.492(c))					
If the written opinion prepared by ISA/US or the international preliminary examination report prepared by IPEA/US indicates all claims satisfy provisions of PCT Article 33(1)-(4)..... \$0				\$	
All other situations..... \$200				<b>200</b>	
23. <input checked="" type="checkbox"/> Search fee (37 CFR 1.492(b))					
If the written opinion of the ISA/US or the International preliminary examination report prepared by IPEA/US indicates all claims satisfy provisions of PCT Article 33(1)-(4)..... \$0				\$	
Search fee (37 CFR 1.445(a)(2)) has been paid on the international application to the USPTO as an International Searching Authority..... \$100					
International Search Report prepared by an ISA other than the US and provided to the Office or previously communicated to the US by the IB..... \$400				<b>100</b>	
All other situations..... \$500					
<b>TOTAL OF 21, 22 and 23 =</b>				<b>600</b>	
<input type="checkbox"/> Additional fee for specification and drawings filed in paper over 100 sheets (excluding sequence listing in compliance with 37 CFR 1.821(c) or (e) or computer program listing in an electronic medium) (37 CFR 1.492(j)). The fee is \$250 for each additional 50 sheets of paper or fraction thereof.					
Total Sheets	Extra Sheets	Number of each additional 50 or fraction thereof (round up to a whole number)	RATE		
<b>20 - 100 =</b>	<b>0 / 50 =</b>	<b>0</b>	<b>x \$250</b>	\$ <b>0</b>	
Surcharge of \$130.00 for furnishing any of the search fee, examination fee, or the oath or declaration after the date of commencement of the national stage (37 CFR 1.492(h)).				\$	
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	\$	
Total claims	<b>26 - 20 =</b>	<b>6</b>	<b>x \$ 50</b>	\$ <b>300</b>	
Independent claims	<b>2 - 3 =</b>	<b>0</b>	<b>x \$200</b>	\$ <b>0</b>	
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			<b>+ \$360</b>	\$ <b>0</b>	
<b>TOTAL OF ABOVE CALCULATIONS =</b>				\$ <b>900</b>	
<input type="checkbox"/> Applicant claims small entity status. See 37 CFR 1.27. Fees above are reduced by 1/2.					
<b>SUBTOTAL =</b>				\$ <b>900</b>	
Processing fee of \$130.00 for furnishing the English translation later than 30 months from the earliest claimed priority date (37 CFR 1.492(i)).				\$ <b>0</b>	
<b>TOTAL NATIONAL FEE =</b>				\$ <b>900</b>	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property				\$ <b>0</b>	
<b>TOTAL FEES ENCLOSED =</b>				\$ <b>900</b>	
				Amount to be refunded:	\$
				Amount to be charged	\$

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

- a. ☐ A check in the amount of \$ \_\_\_\_\_ to cover the above fees is enclosed.
- b. ☐ Please charge my Deposit Account No. \_\_\_\_\_ in the amount of \$ \_\_\_\_\_ to cover the above fees.  
A duplicate copy of this sheet is enclosed.
- c. ☐ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. \_\_\_\_\_. A duplicate copy of this sheet is enclosed.
- d. ☒ Fees are to be charged to a credit card. **WARNING:** Information on this form may become public. Credit card information should not be included on this form. Provide credit card information and authorization on PTO-2038.

NOTE: Where an appropriate time limit under 37 CFR 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the International Application to pending status.

SEND ALL CORRESPONDENCE TO:

Kevin D. McCarthy  
Roach Brown McCarthy & Gruber, P.C.  
420 Main St. - 1620 Liberty Bldg.  
Buffalo, NY 14202

SIGNATURE

NAME

REGISTRATION NUMBER

# Exhibit D





## UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE  
 United States Patent and Trademark Office  
 Address: COMMISSIONER FOR PATENTS  
 P.O. Box 1450  
 Alexandria, Virginia 22313-1450  
 www.uspto.gov

U.S. APPLICATION NUMBER NO.

10/588,398

FIRST NAMED APPLICANT

Samuel Bron

ATTY. DOCKET NO.

0-06-172

INTERNATIONAL APPLICATION NO.

PCT/IL05/00554

I.A. FILING DATE

05/30/2005

PRIORITY DATE

06/10/2004

Kevin D. McCarthy  
 Roach Brown McCarthy & Gruber, PC  
 1620 Liberty Building  
 420 Main Street  
 Buffalo, NY 14202

CONFIRMATION NO. 8382

371 FORMALITIES LETTER



\*OC000000022552944\*

Date Mailed: 02/20/2007

### NOTIFICATION OF MISSING REQUIREMENTS UNDER 35 U.S.C. 371 IN THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US)

The following items have been submitted by the applicant or the IB to the United States Patent and Trademark Office as a Designated / Elected Office (37 CFR 1.495).

- Copy of the International Application filed on 08/03/2006
- Copy of the International Search Report filed on 08/03/2006
- Preliminary Amendments filed on 08/03/2006
- Information Disclosure Statements filed on 08/03/2006
- Oath or Declaration filed on 11/14/2006
- Request for Immediate Examination filed on 08/03/2006
- U.S. Basic National Fees filed on 08/03/2006
- Substitute Specification filed on 08/03/2006
- Priority Documents filed on 08/03/2006
- Specification filed on 08/03/2006
- Claims filed on 08/03/2006
- Abstracts filed on 08/03/2006

The following items **MUST** be furnished within the period set forth below in order to complete the requirements for acceptance under 35 U.S.C. 371:

- Oath or declaration of the inventors, in compliance with 37 CFR 1.497(a) and (b), identifying the application by the International application number and international filing date. The current oath or declaration does not comply with 37 CFR 1.497(a) and (b) in that it:
  - The 5th through 9th inventors were not listed on the cover page of the International Application, as inventors for the U.S. Please submit a corrected declaration or submit the appropriate petition papers (if attempting to add inventors).

**ITEMS SET FORTH ABOVE MUST BE SUBMITTED WITHIN TWO (2) MONTHS FROM THE NOTICE OR BY 32 MONTHS FROM THE PRIORITY DATE FOR THE APPLICATION,**